# PATENT SPECIFICATION

DRAWINGS ATTACHED

**989,424**

989,424

*Date of Application and filing Complete Specification: Sept. 29, 1961.*

*No. 35121/61.*

*Application made in Italy (No. 17497) on Oct. 5, 1960.*

*Complete Specification Published: April 14, 1965.*



**Index at acceptance:—B1 X16**

**Int. Cl.:—B 01 j**

COMPLETE SPECIFICATION

## Method and apparatus to Effect Reactions in an Automatic Manner

I, MARIO BALLESTRA, of Viale Bianca Maria, 26, Milan, Italy, of Italian nationality, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method and apparatus for effecting reactions automatically between at least two reagents, one of which is in the gaseous or vapour phase.

In the chemical industry the case often occurs when it is necessary to effect reactions in a discontinuous manner, as the type of reaction does not allow for the use of suitable means to make it continuous.

In such cases it is important to be ready to proceed rapidly with the successive batches in order to exploit the equipment to its maximum, thus rendering the method practically continuous. But it is also necessary to simultaneously remove the reaction product from the reactor, only when it is sure that the reaction has been completely effected.

Among the various types of reaction included in the above case, is the one in which the reaction involves at least one reagent in the gaseous or vapour phase and the reaction product comprises at least one component in the gaseous or vapour phase.

The invention consists in a method of effecting reactions in an automatic manner wherein in addition to at least one reactant in the gaseous or vapour phase there is one liquid reactant, and wherein the reaction container is fed first with a previously proportioned amount of the liquid reactant and then with the gaseous reactant, the feeding of this reactant in the gaseous or vapour phase to the reaction container is automatically terminated by a weighing device on which acts the total weight of the reactor and its contents.

The invention also consists in an apparatus for carrying out the method described in the preceding paragraph wherein the reactor is arranged on a weighing device sensitive to the weight of the reactor and of the material contained therein, the weighing device being provided with means suitable to transmit automatically the control for opening or closing the feeding valve of the gaseous reactant when the weight measured by the weighing device reaches predetermined values.

The invention will now be described, by way of example only with reference to the accompanying drawings, wherein,

Figure 1 is a general diagrammatic view of an apparatus comprising two reactors designed to work automatically and continuously.

Figure 2 is a modification showing a particular case.

With particular reference to Figure 1 a feeding piping 1 for the liquid reactant, has an electromagnetic valve 2 controlling the inlet of liquid reagent into a feed proportioning vessel 3, the valve 2 being controlled by a weighing device 4 which is actuated by the weight of the feed proportioning device 3 and contents. Discharge piping 5 for the liquid reagent proportioned in the proportioning vessel 3 carries the reactants alternatively to reactors 8 and 9. Two electromagnetic valves 6 and 7, controlled by weighing devices 12 or 13, are arranged on the discharge piping 5 for the liquid reactant proportioned in the vessel 3 to be fed into the reactor containers 8 and 9. These reactor containers are each provided with a heating and/or cooling coil *a*. Within the containers 8 and 9 stirrers 10 and 11 are provided, while the containers 8 and 9 are supported on the weighing devices 12 and 13. The gaseous reactant is fed through pipe 14. Electromagnetic valves 15 and 16 controlling the inlet of the gaseous reactant into the reactor containers 8 and 9 are actuated by the weighing devices 12 and 13 which thus control the weight of the contents of reactor containers 8 and 9. Valves 24 and 25 controlling the

discharge of the two reactor containers 8 and 9 may be electromagnetic valves controlled by the indication of the weighing devices 12 and 13. 26 is a discharge piping of the reactor containers.

The working of the apparatus shown in Figure 1 is as follows:—

The reactant fed from piping 1 through the valve 2 is introduced into the proportioning vessel 3, and the predetermined amount of liquid reactant, the weight of which is controlled by the weighing device 4, automatically closes the valve 2 when the amount of liquid fed to the vessel 3 has reached a given weight. The opening of valves 6 or 7, in the way described herebelow, causes the discharge of the vessel 3, therefore the weighing device 4 will make the valve 2 open when the weight of the vessel 3 has reached a minimum value corresponding to its complete discharge. The operation of reactor 8 subsequently takes place in three phases, charge of the liquid reactant, charge of the gaseous or vapour reactant and discharge of the reaction product. That is:

a) charge of the liquid reactant through the valve 6 which is open while valves 15 and 24 are closed.

b) charge of the reactant in the gas or vapour phase through the valve 15, while the valves 6 and 24 are closed.

c) discharge of the reaction product through the valve 24, while valves 6 and 15 are closed.

The valves 6 and 24 may be controlled and made to open and close by means of the weighing device 12. In the latter case when the outlet of the reaction products from the reactor 8 is open, the weight of the reactor 8 reaches a minimum value in correspondence to which the weighing device 12 makes the valve 24 close and the valve 6 open at the same time, while valve 15 remains closed. The reactor 8 is then charged with the liquid reactant. When the pre-proportioned amount of liquid reactant has completely reached the reactor 8, from the vessel 3, the weight of the reactor and its contents reaches a given weight in correspondence of which the weighing device 12 makes the valve 15 open and the valve 6 close, while the valve 24 remains closed. At this point the gaseous reactant flows to the reactor 8, passing through the mass of liquid reactant, with which it combines and mixes forming a reaction product, the weight of which is different compared to the weight of the liquid reactant alone. When the mass of gaseous reactant, introduced into the reactor 8, has made all the liquid reactant react, the weight of the reactor 8 and its contents reaches a given weight in correspondence of which the weighing device 12 makes the valve 15 close, the valve 24 open, while the valve 6 remains closed. At this point, the working cycle of the reactor 8 starts again.

The operation of the valves 7, 16 and 25 of the reactor 9 resting on the weighing device 13 is the same as the valves 6, 15 and 24, but it is staggered with respect to the latter ones in such a way that, while, for instance, the gaseous reagent is introduced through the open valve 15 and reacts inside the reactor 8, at the same time the valves 6 and 24 are closed, the second reactor 9 is first emptied of the liquid products of the previous reaction and is then fed with the previously proportioned amount of liquid reagent. The second reactor 9 will then be ready to receive the inflow of gaseous reagent, from the valve 16, while the first reactor 8 will start the discharge step of the liquid products of the reaction.

It is obvious that whilst the equipment may be provided with only two reactor containers 8 and 9, additional proportioning vessels 3 may be employed in the case where there are more than one liquid reactant involved in the reaction.

Figure 2, in which like parts have the same references as in Figure 1, deals with a method of sulphonation of a material coming through valve 2. A water feeding piping 17 has a valve 18, controlling the inlet of the water into a feed proportioning device 19, and is actuated by the feeding controlling device consisting of a siphon 20. Discharge piping 21 from the water proportioning device 19 leads to electromagnetic valves 22 and 23, or controlled by the weighing devices 12 and 13 for feeding water into the two reactors 8 and 9. In a subsequent neutralisation step, an inlet piping 27 for the neutralising reactant is controlled by a valve 28 and is fed into a proportioning device 29, said valve being actuated by a weighing device consisting of a scale 30. An electromagnetic valve 31 controlling the inlet of the neutralizing reactant into a neutralizing container 32 is connected with a proportioning pump 35. The container 32 is provided with a heating or cooling coil 36 and an agitating device 33. The valve 31 may be actuated by a pH-meter which controls the pH value within the container 32. Container 32 has a bottom discharge valve 34 and a discharge overflow 37. Incorporated in the pipe from the proportioning devices 8 and 9 is a container 38 for the product obtained in the reactors 8 and 9, also being connected to a proportioning pump 39. The container 38 comprises an intermediate vessel having the function of a tank in order to make regular the working of pump 39. The discharge pipe 34 is used for a discontinuous process, and the overflow discharge pipe 37 for a continuous process. The apparatus shown in Figure 2 works as follows:—

It is to be noted that the apparatus shown in Figure 2 is particularly suitable for sulphonation with sulphur trioxide. The material to be sulphonated (e.g. alkylbenzenes, alcohols, sulphonates and olefinic products) is introduced through the piping 1 into the proportioning

container 3 through the valve 2 which automatically closes as soon as the weight controlled by the scale corresponds to the predetermined amount for the load of the first sulphonation container 8.

The proportioned amount then passes through piping 5 and the valve 6 which may be actuated by the weighing device 12 into the first sulphonation reactor 8. Valve 6 is automatically shut and valve 15 opened thus introducing the sulphur trioxide which may be sulphur trioxide evaporated and mixed with air so as to obtain a mixture containing about 4% to 8% of sulphur trioxide.

Conveniently, mixtures of sulphur trioxide, with a content of oxygen lower than that of air so as to prevent the sulphonated product from oxidizing and darkening are used. Small amounts of gaseous reducing agents, in particular sulphur dioxide, may be also added.

A further advantage is obtained by using directly the gases obtained by catalytic conversion of sulphur dioxide to sulphur trioxide, said gases being previously cooled. Such a gas has in fact a low content of oxygen and further contains small amounts of sulphur dioxide acting as a reducer and thus as a bleaching agent for the product.

The mixture containing gaseous sulphur trioxide is introduced into the first reactor 8 from the bottom near the agitating device 10. On the scale of the weighing device 12 is established the weight which the sulphonated product will have, so as to automatically stop the flow of sulphur trioxide gases as soon as the predetermined weight is reached.

For instance, while an alkylbenzene has a specific weight of about 0.870, an alkylbenzene sulphonic acid has a specific weight of about 1.960, so that when the weight corresponding to this specific weight is reached the weighing device automatically shuts the electromagnetic valve 15. When the sulphonation is ended, there is introduced into the reactor 8 a predetermined amount of water proportioned in the proportioning device controlled by the siphon 20 or by another device controlling the weight or the volume. Valve 22, for instance controlled by a weighing device, introduces into the reactor 8 the predetermined amount of water. Valve 24, for instance controlled by a weighing device, is then open to discharge the sulphonated product into the container 32 for the neutralization.

While the sulphonation takes place within the reactor 8, the reactor 9 is discharged of the liquid products of the preceding reaction and thus the proportioning device 3 charges the reactor 9 with a liquid reagent and, as soon as the reaction within the reactor 8 is over, the valve 16, controlled by the weighing device 13 is opened so that the reactions within the reactors 8 and 9 follow automatically and rapidly after one another, exploiting the equipment to its maximum output and above all allowing the flux of gaseous reagent to flow continuously inside conduit 14 to the direction of one or the other of the reactors 8 and 9 alternately.

In the container 32 from the piping 26 through the container 38 and the proportioning pump 39 the sulphonated product is fed, and from the piping 27, through the proportioning device 29 mounted on the scale 30 controlling the inlet valve and through the valve 31 controlled by a weighing device or by a pH-meter, the neutralization agent e.g. sodium hydroxide is fed.

The neutralization may take place in a discontinuous manner, by feeding the sulphonic acid through a direct connection (not shown in the figure) between the valve 25 and the neutralizing container 32, and by feeding the neutralizing agent directly from the proportioning device 29 and the valve 31, discharging the neutralized product through the bottom valve 34. The neutralization may even be effected in a continuous manner by discharging the sulphonic acid into the container 38 and feeding the container 32 by means of a proportioning pump 39, while the neutralizing agent is fed to the neutralizing device through the proportioning pump 35 and the neutralized product is discharged from the neutralizing container 32 in a continuous way by overflow through the conduit 37.

Although the present invention has been described with reference to two preferred embodiments many changes, variations, and additions, may be made to both the method and the apparatus for example by providing within the reactors 8 and 9 suitable breathers allowing the removal of any gas or gases and/or vapours developing during the reaction, or of the excess of the gaseous reactants.

WHAT I CLAIM IS:—

1. A method of effecting reactions in an automatic manner wherein in addition to at least one reactant in the gaseous or vapour phase there is one liquid reactant, and wherein the reaction container is fed first with a previously proportioned amount of the liquid reactant and then with the gaseous reactant, the feeding of this reactant in the gaseous or vapour phase to the reaction container is automatically terminated by a weighing device on which acts the total weight of the reactor and its contents.

2. A method as claimed in claim 1, wherein the weighing device is calibrated by known means to control the valves for the introduction of the reactant in the gaseous or vapour phase, and further to control the valve for the discharge of the reactor, when the weight of the reactor and its contents reaches a given weight corresponding to the one needed to show that the reaction is completed.

3. A method as claimed in claims 1 or 2, wherein the reactants are alternately introduced into a second reaction container, so that the

flow of the gaseous reagent directed alternately to one or the other of the two reactors is continuous.

4. A method as claimed in any of claims 1 to 3, wherein for its special use for effecting sulphonation reactions, gaseous sulphur trioxide is suitably mixed with a gas having a low content of oxygen and containing small quantities of a gas with a reducing action.

5. A method as claimed in claim 4, wherein the mixture of sulphonation gases containing sulphur trioxide comes directly from the catalytic conversion of sulphur dioxide to sulphur trioxide.

6. An apparatus for carrying out the method as claimed in any of claims 1 to 5, wherein the reactor, is arranged on a weighing device sensitive to the weight of the reactor and of the material contained therein, the weighing device being provided with means suitable to transmit automatically the control for opening or closing the feeding valve of the gaseous reactant, when the weight measured by the weighing device reaches a predetermined value.

7. An apparatus as claimed in claim 6, wherein the weighing device sensitive to the weight of the reactor and of the material contained therein, is provided with means suitable to transmit the control for opening or closing the feeding valve of the liquid reactant and the discharge valve of the product of the reaction from the reactor, when the weight measured by said sensitive weighing device reaches a predetermined value.

8. An apparatus as claimed in claims 6 or 7, wherein the means suitable to transmit the control for opening and closing the valves of the apparatus itself in correspondence to a predetermined value of the weight of the reactor and of the material contained therein:

a) close the discharge valve from the reactor and open the feeding valve of the liquid reactant, while simultaneously the feeding valve of the reactant in the gaseous or vapour phase is closed, when the weight of the reactor and of the material contained therein reaches a minimum value corresponding to the complete discharge of the reaction compounds, from the reactor.

b) close the feeding valve of the liquid reactant and open the feeding valve of the reactant in the gaseous or vapour phase, while the discharge valve of the reactor remains closed, when the weight of the reactor and of the material contained therein reaches the value corresponding to the presence inside the reactor of the amount of liquid reactant predetermined for each reaction.

c) close the feeding valve of the reactant in the gaseous or vapour phase and open the discharge valve of the reaction products from the reactor, while the feeding valve of the liquid reactant remains closed, when the weight of the reactor and of the material contained therein reaches the value corresponding to the one needed to show that the reaction is completed, that is when the liquid reactant has completely transformed into the reaction product owing to the action of the reactant in the gaseous or vapour phase.

9. A method of effecting a reaction in an automatic manner, substantially as hereinbefore described with reference to the accompanying drawings.

10. An apparatus for carrying out the method claimed in claim 9, substantially as hereinbefore described with reference to and as shown by the accompanying drawings.

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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1965. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.

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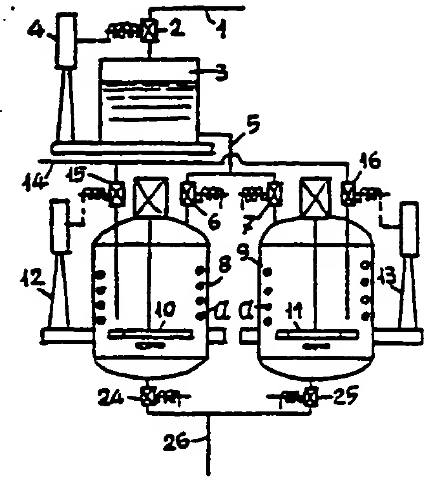


Fig. 1

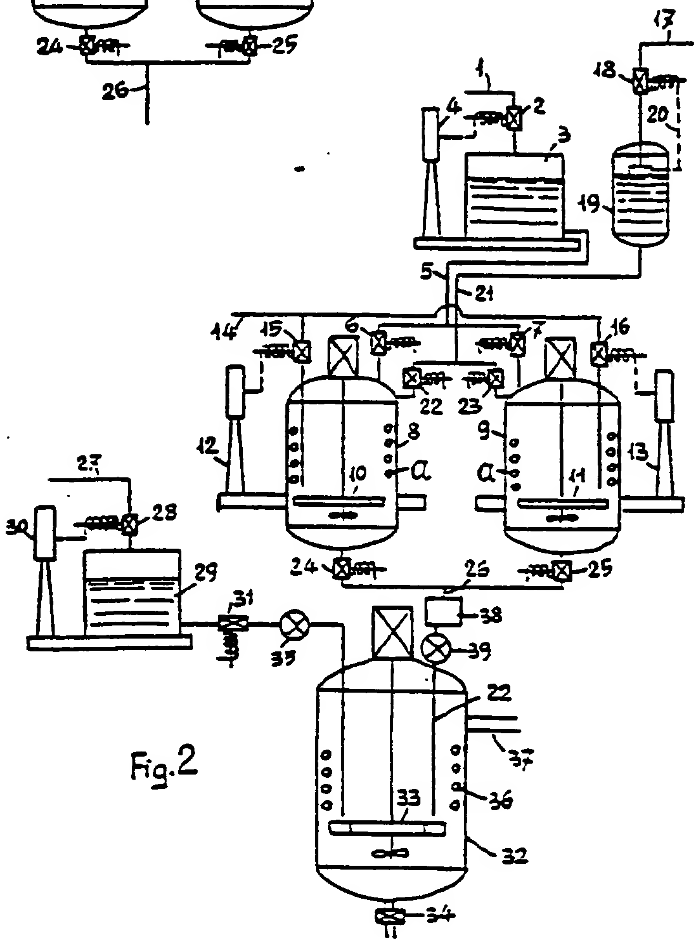


Fig. 2